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COUNTRY Germany (Federal Republic)

SUBJECT Controlled, Organ Specific Chemotherapy in Prostate Carcinoma [redacted]

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PLACE ACQUIRED - - -
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DATE OF INFO

Guide 27
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This UNEVALUATED INFORMATION is supplied for the possible interest of your analysts. It does not warrant dissemination by [redacted]

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He translated, at the request of his superiors, the attached paper entitled "Controlled, Organ Specific Chemotherapy in Prostate Carcinoma", which was originally written in German by Dr. H. Wilmanns of the Research Department of the Asta-Werke A.G., Brackwede, Westphalia, Germany.

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UNLESS OTHERWISE SPECIFIED
TOLERANCE LIMITS
ON FRACTIONAL DIMENSIONS ±
ON DECIMAL DIMENSIONS ±
ON ANGULAR DIMENSIONS ±
ON CONCENTRICITY
USED ON MODELS

DR BY H.E.B.
DATE 1-27-54
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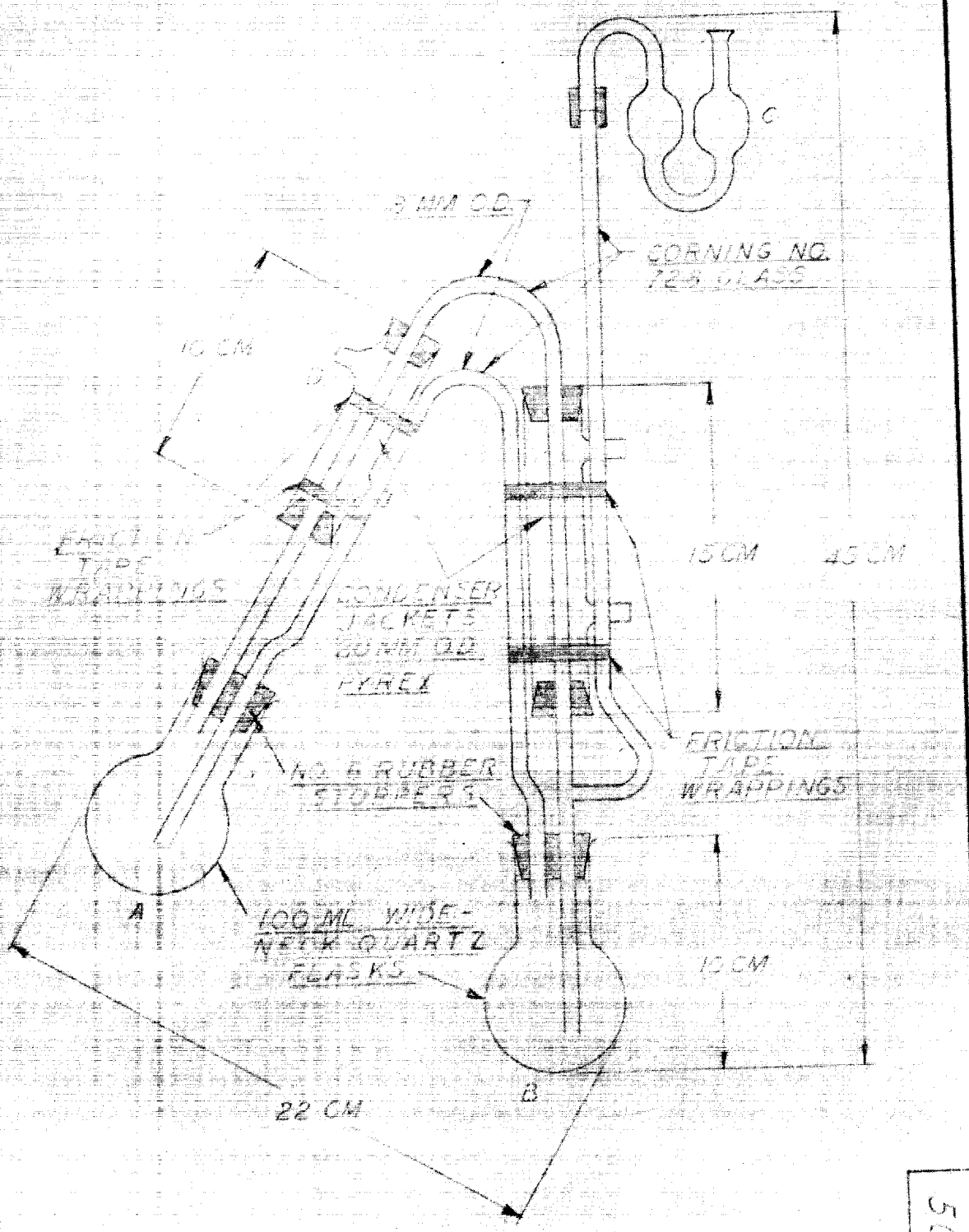
SCALE NONE
MATERIAL NOTED

NAME BORON DISTILLATION APPARATUS

EXP. PART NO.

PROD. PART NO. 503-20



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BORON BY THE DISTILLATION - CURCUMIN (PHOTOMETRIC) METHOD

(For Plain Carbon Steels Containing Under 0.008% Boron)
from pp. 135-143 of 1950 Book of ASTM Methods for Chemical Analysis of Metals

Principle of Method

Boron is separated by distillation as methyl borate. The isolated boric acid reacts with curcumin to form a rose-colored compound. The photometric measurement is made at approximately 540 mμ.

Concentration Range

The recommended concentration range is from 0.001 to 0.008 mg. of boron in 100 ml. of solution, using a cell depth of 2 cm. [1]

Stability of Color

The color is stable for about 1 hr.

Interfering Elements

The elements ordinarily present in steel do not interfere. The analyst should exercise care to see that phosphoric acid does not spray over during the solution or distillation of the sample, as phosphoric acid will bleach the color.

Apparatus

(a) Apparatus for Determination of Boron by Distillation - see Appendix.
(b) Casseroles - Porcelain casseroles of 300-ml. capacity, preferably new.
(c) Water Bath - An automatically controlled water bath capable of maintaining a temperature of 55 ± 3C.
(d) Filtering Crucible - A fritted-glass crucible of fine porosity.

Reagents [2]

(a) Standard Boric Acid Solution (1 ml. = 0.002 mg. B). - Transfer 0.572 g. of H_3BO_3 to a 500-ml. volumetric flask, dilute to the mark with freshly distilled water, and mix. Transfer 10 ml. of this solution to a 1-liter volumetric flask, dilute to the mark with freshly distilled water, and mix. Store in a low-boron container.
(b) Calcium Hydroxide Suspension (5.6 g. CaO per l.). - Ignite low-boron $CaCO_3$ (0.0001 per cent or less of boron) in a platinum dish at 500 to 600 C. Gradually raise the temperature to 1000 C. and hold at this temperature for at least 30 min. Cool, and grind in an agate mortar. Add 5.6 g. of the finely ground CaO to 1 liter of freshly distilled water, and store in a low-boron container. Thoroughly mix the solution each time before drawing off a portion for use in analysis.

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1. This procedure has been written for a cell having a 2-cm. light path. Cells having other dimensions may be used, provided suitable adjustments can be made in the amounts of sample and reagents used.

2. All solutions and reagents shall be stored in bottles made of low-boron glass.

(c) Low-Boron Steel.[3]
(d) Methanol. - Add 0.5 g. of NaOH pellets to 0.5 liter of absolute methanol, and distill in low-boron apparatus. Store in a low-boron container.
(e) Phosphoric Acid (85%) - The boron content of 10 ml. of the H_3PO_4 shall not exceed 0.001 mg.[4]
(f) Oxalic Acid Solution - Dissolve 50 g. of oxalic acid in 450 ml. of acetone and filter. This solution is stable for approximately two weeks.
(g) Curcumin Solution (0.25 g. per l.) - Dissolve 0.10 g. of curcumin in 400 ml. of ethanol (95%), filter,[5] and store, preferably in a brown low-boron bottle. This solution should be kept not longer than four weeks. One or two points on the calibration curve should be checked each time a new solution is prepared.
(h) Acetone - Low-boron acetone.

Preparation of Calibration Curve

(a) Transfer 1.0, 2.0, 3.0, and 4.0-ml. aliquots of boric acid (1 ml. = 0.002 mg.B) to 100-ml. quartz flasks A (see drawing in appendix). To each flask and to an additional flask to be carried through as a blank, add 5 ml. of $Ca(OH)_2$ suspension (5.6 g. CaO per l.).
(b) Evaporate the contents of the flasks to dryness. Add 0.5 g. of low-boron steel to each flask, including the blank.
(c) To a 100-ml. quartz flask B, add 50 ml. of methanol and 5 ml. of $Ca(OH)_2$ suspension (5.6 g. CaO per l.). To the trap C, add enough $Ca(OH)_2$ suspension (5.6 g. CaO per l.). to form a liquid seal. Add 10 ml. of H_3PO_4 to the flask A containing the sample and assemble the apparatus as illustrated in the drawing. Turn on the water supply to both the auxiliary condenser D and the condenser leading to flask B, and heat the flask containing the sample gently by means of a small burner until the reaction ceases. Remove the heat from the flask, and disconnect the water supply from the auxiliary condenser D.
(d) Place flask B in the hot water, and heat until about 25 ml. of methanol has distilled over into flask A. Then place both flasks in hot water, and heat so that the methanol will cycle evenly between flask A and flask B for 30 min.
(e) Remove the flasks from the water baths and transfer the solution from flask B and from the trap C to a 300-ml. porcelain casserole. Rinse the flask and trap thoroughly, first with water, then with two drops of HCl (1:9), and again with water, adding all of the washings to the casserole. Evaporate to dryness on a steam bath, remove, and cool to room temperature.
(f) To the residue in the casserole, add 1 ml. of HCl (1:4) and 5 ml. of oxalic acid solution, mix, and add 2 ml. of curcumin solution (0.25 g. per l.). When the residue in the

3. National Bureau of Standards standard sample No. 55 of ingot iron is satisfactory for this purpose.
4. Low-boron analytical reagent grade H_3PO_4 (85%) has been found satisfactory for this purpose.
5. Certain filter papers contain alcohol-soluble boron; hence, it

casserole has dissolved, evaporate to dryness on a water bath at a temperature of 55 ± 3 C. and bake for 30 ± 5 min. at the same temperature.

(g) To the residue in the cooled casserole, add 25 ml. of acetone. After the residue has dissolved, filter through a fritted-glass crucible of fine porosity into a 100-ml. volumetric flask. Wash the crucible and contents with 25 ml. of acetone, using 3- to 5-ml. portions for each washing. Dilute the solution to 100 ml. with cold water and mix well. The color will vary from a yellowish green to a deep rose, depending upon the boron concentration.

(h) Transfer a suitable portion of the solution to an absorption cell, and measure the absorbancy or transmittancy at approximately 540 mμ. Compensate or correct for the blank.

(i) Plot the values obtained against milligrams of boron per 100 ml. of solution.

Procedure

(a) Transfer to a 100-ml. quartz flask A, 0.5 g. of the sample for a steel containing 0.002 per cent or less of boron, 0.25 g. for a steel containing 0.002 to 0.004 per cent of boron, or 0.10 g. for a steel containing 0.004 to 0.008 per cent of boron. Transfer to another 100-ml. quartz flask a weight of low-boron steel equal to the weight of sample taken, and carry through all steps of the procedure.

Acid-Soluble Boron

(b) Proceed as directed in sections (c) to (h) under "Preparation of Calibration Curve", reserving the flask A (section (e)) for the determination of acid-insoluble boron.

(c) Using the value obtained, read from the calibration curve the number of milligrams of boron present in the sample.

(d) Calculation - Calculate the percentage of acid-soluble boron as follows:

$$\text{Acid-soluble boron, per cent} = \frac{A}{B \times 10}$$

where:

A = milligrams of boron (section (c)), and

B = grams of sample used.

Acid-Insoluble Boron

(e) Dilute the solution in flask A, reserved as directed in section (b), to a volume of 90 ml. with HCl (1:8). Filter through a 9-cm., close-texture, ashless paper containing

with hot HCl (2:98) and police the flask well to remove all insoluble material. Wash the residue on the filter paper,

first with hot HCl (2:98) to remove the iron, and then with cold water to remove the HCl.

(f) Transfer the paper and residue to a 15- to 20-ml. platinum crucible. Add 5 ml. of $Ca(OH)_2$ suspension (5.6 g. CaO per l.), and evaporate to dryness. Ignite at 600 to 700 C. until the carbon is completely burned. Add 1 g. of Na_2CO_3 and fuse the residue, finally tilting and heating the crucible so that the fusion is collected in a ball. Cool, remove the greater part of the fused mass by exerting a small amount of pressure on the crucible wall, and transfer the fusion to a clean, dry quartz flask A. Cool to 10 to 15 C. by placing the flask in cool water. Add 4 ml. of H_3PO_4 to the crucible, warm to dissolve the remainder of the fusion, cool and add to flask A. Rinse out the crucible with two 3-ml. portions of H_3PO_4 and add to flask A. Assemble the apparatus immediately as directed in section (c) of "Preparation of Calibration Curve". Heat flask A to obtain complete solution of the fusion, and continue as directed in paragraphs (b) and (c) of this section.

(g) Calculation - Calculate the percentage of acid-insoluble boron as follows:

$$\text{Acid-insoluble boron, per cent} = \frac{A}{B \times 10}$$

where:

A. = milligrams of boron (section (f)), and

B = grams of sample used.

Total Boron

(h) Total boron equals the percentage of acid-soluble boron plus the percentage of acid-insoluble boron.

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APPENDIX

The apparatus shall be suitable for the distillation of methyl borate from a phosphoric acid solution of steel. A typical arrangement is shown in the attached drawing. Flasks A and B shall be 100-ml., wide-neck quartz flasks. The remainder of the apparatus, including the traps, (with the exception of the condenser jackets) shall be constructed of low-boron glass.

the above apparatus is available through

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CIA, [redacted]

GERMANY (FR) 1 SEP 54
CONTROLLED, ORGAN SPECIFIC CHEMO-
THERAPY IN PROSTATE CARCINOMA.
(INFO JUNE 1954)
IP [redacted]TD/US ONLY
1 ENCL. - IIP (TRANSL. OF PAPER SAME AS
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